

The author wishes to thank Dr. Fitzgerald Dunning, of Hynson, Westcott and Dunning, Inc., for the use of their laboratories and material while carrying out this investigation.

SUMMARY.

p-Butyl saligenin has been prepared from *p*-butyl phenol by the formation of 5-butyl salicylaldehyde and its subsequent reduction with hydrogen.

BIBLIOGRAPHY.

- (1) Sandulesco and Girard, *Bull. soc. chim.* (4), 47 (1932), 1300.
- (2) Read and Mullin, *J. Am. Chem. Soc.*, 50 (1928), 1763.
- (3) Read and Foster, *Ibid.*, 48 (1926), 1606.
- (4) Radziszewski, *Ber.*, 9 (1899), 261.
- (5) Balbino, *Ibid.*, 10 (1900), 296.
- (6) Smith, Richard A., *J. Am. Chem. Soc.*, 56 (1934), 1419.
- (7) Reimer and Tiemann, *Ber.*, 9 (1899), 824; 10 (1900), 213.
- (8) Adams, Voorhees and Shriner, "Organic Syntheses," Vol. VIII (1928), p. 92.
- (9) Perkin, *J. Chem. Soc.*, 55 (1889), 548.

BISMUTH-SODIUM-POTASSIUM TARTRATE SOLUTIONS.*

BY A. H. CLARK.¹

Considerable interest has been taken during recent years in the so-called complex bismuth tartrates. The soluble salts have been used extensively in the treatment of syphilis in place of arsenic compounds, and solutions have been used for local applications. This communication deals with the preparation of a neutral solution containing bismuth in combination with tartrate and glycerin.

After many experiments or trials it has been established that such solutions are readily obtained by the procedure outlined:

Bismuth hydroxide is first prepared as free as possible from bismuth salts by dissolving bismuth subnitrate or subcarbonate in water and nitric acid. This solution is then precipitated with ammonia water and washed by decantation and collected by aid of a Büchner filter and moisture removed as completely as possible by suction.

The moist precipitate is then added to glycerin and a solution of Rochelle salt. To this mixture is then added sodium or potassium hydroxide until solution takes place.

To the strongly alkaline solution thus obtained is added tartaric acid until the mixture is neutral to litmus paper.

By this procedure a solution is obtained that is neutral or faintly acid to litmus or phenolphthalein and shows slight alkalinity to methyl orange. It will also contain a small amount of nitrate. Just what is the state of combination between the tartrate, bismuth and glycerin it is impossible to state as the writer has prepared a large number of solutions in which the proportions of the three vary widely. It is possible to vary the proportions of bismuth, glycerin, alkali and tartrate within wide limits. Indeed it is possible to prepare solutions from bismuth hydroxide, glycerin and alkali alone but such solutions are strongly alkaline in reaction. By starting with a stated amount of bismuth salt yielding a known per cent of Bi_2O_3 upon ignition, a solution containing a desired per cent of Bi_2O_3 may be

* Scientific Section, A. P. H. A., Portland meeting, 1935.

¹ University of Illinois, College of Pharmacy, Chicago.

readily obtained. A typical procedure is as follows, the quantities given yielding a solution containing about 12 Gm. Bi_2O_3 in 100 cc.:

Bismuth Subcarbonate	15 Gm.
Water	25 cc.
Conc. nitric acid	25 cc.

Heat, but do not boil, until solution is complete and all carbon dioxide is expelled. Dilute to about 600 cc. but not to the precipitation point. Add ammonia water in slight excess. Use litmus paper and have the mixture alkaline throughout. Collect the precipitate on a Büchner filter by suction, washing well to remove soluble salts.

Dissolve 10 Gm. Rochelle salt in water to make about 25 cc. When the Rochelle salt is dissolved add 20 cc. of glycerin and heat to about 100° C. Do not boil.

To the hot mixture add the bismuth precipitate and mix well. To this mixture add a 50% solution of sodium hydroxide, a few drops at a time, stirring continuously until solution is complete; to this solution add a pasty mixture of tartaric acid and water, a little at a time, until it just turns blue litmus red. No precipitation should occur at this point, but if it does—a drop or two of ammonia will usually clear up the precipitate. Add water to bring the volume up to 100 cc.

The above has been varied by increasing or decreasing the glycerin, substituting sodium tartrate or potassium tartrate for the Rochelle salt, potassium hydroxide for the sodium hydroxide, etc. It has been found that within a quite liberal range all these factors may be varied and solutions obtained that seem to be permanent.

Experiments are under way to determine whether or not a solid bismuth compound that is permanent and soluble in water can be separated from such solutions.

DRUG EXTRACTION. VI. DETERMINATION OF THE PRESSURE EXERTED BY A DRUG DURING PERCOLATION.*¹

BY WILLIAM J. HUSA² AND LOUIS MAGID.

Previous experiments (1) seem to indicate that maximum swelling of powdered drugs is not attained with the proportions of liquid ordinarily used in moistening drug powders preparatory to packing in a percolator. From this it would follow that a certain amount of pressure would be developed in the percolator after the drug has been packed and excess menstruum added. This pressure might affect the imbibition of the menstruum, the solvent power of the menstruum, or result in a slowing or even complete stopping of the process of percolation. Because of the fundamental importance of this question, an apparatus and a method have been developed for the determination of the pressure exerted by a drug during percolation. Using this new apparatus, measurements have been made on powdered belladonna root, rhubarb, senna and red cinchona.

APPARATUS FOR DETERMINATION OF PRESSURE.

An apparatus (Fig. 1) has been developed, whereby the pressure exerted by a powdered drug during percolation can be measured. A rubber tube, closed at one end and connected to

* Scientific Section, A. Ph. A., Portland meeting, 1935.

¹ This investigation was aided by a grant from the AMERICAN PHARMACEUTICAL ASSOCIATION Research Fund.

² Head Professor of Pharmacy, University of Florida.